Investigation of Alternative Materials for Impregnation of Nb₃Sn Magnets

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Abstract—Insulation is one of the most important elements of magnet design, which determines the electrical, mechanical, and thermal performance as well as lifetime of the magnet. The exposure to high radiation loads especially for the proposed LHC second-generation interaction region Nb₃Sn quadrupoles further limits the choices of the insulation materials. Traditionally Nb₃Sn magnets were impregnated with epoxy to improve both the mechanical and electrical properties. However, the acceptable radiation limit for epoxy is low which reduces the lifetime of the magnet. The paper presents the results of the feasibility study to replace epoxy with high radiation-resistant material during vacuum impregnation. The mechanical, thermal and electrical properties of samples impregnated with Matrimid were measured and compared with epoxy-impregnated samples.

Index Terms—Radiation resistance, polyimides, epoxy, Nb₃Sn.

I. INTRODUCTION

THE first generation of low-β quadrupoles for LHC inner triplet based on NbTi superconductor technology are being produced at Fermilab and KEK [1,2]. Based on the radiation dose, the estimated lifetime of these magnets at nominal luminosity of 10³⁴ cm⁻²s⁻¹ is about 6–7 years. Note that this estimate is in the high luminosity interaction regions. The limiting factor is the radiation strength of coil end-parts, which consist of substantial fraction of epoxy. The planned luminosity upgrades [3] will further reduce the lifetime of the magnets in the high luminosity regions to about 3 years.

In order to reach the highest possible machine luminosity without compromising the lifetime of the magnets, a new generation of quadrupoles made from superconductors with higher critical parameters than NbTi and structural materials with radiation strength higher than that of G11 are being investigated. Nb₃Sn is proposed as a choice of superconductor for 2nd generation LHC IR quadrupoles [4]. The lifetime of a magnet based on Nb₃Sn superconductor technology will depend on the choice of coil end-part material, cable insulation and the material used during vacuum impregnation. Since the coil end-parts are made from aluminum bronze and ceramic tape is used as cable insulation [5], the material used during vacuum impregnation will determine the lifetime of a

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Nb₃Sn magnet. Traditionally these magnets are impregnated with epoxy to improve both the mechanical and electrical properties of the coil. However, the acceptable radiation dose for epoxy is quite low. The paper presents the results of the study currently underway at Fermilab to replace epoxy with high radiation-resistant material like polyimides / bismaleimides.

II. MATERIALS INVESTIGATED

Various polyimide/bismaleimides that are commercially available were investigated to replace epoxy as a medium of impregnation for Nb₃Sn magnets. The determining factors for the applicability of these solutions are the viscosity and potlife. An ideal solution for vacuum impregnation will have low viscosity and long potlife. Note that the viscosity of most of the available polyimide solutions is reduced through the addition of solvents. However, these solutions cannot be used under vacuum due to out-gassing. On the other hand, solventless polyimide solutions usually have high viscosity at room making them unsuitable for vacuum impregnation. However, raising the temperature, the upper limit of which is determined by the potlife, can reduce viscosity. Note that the potlife is inversely proportional to the temperature.

A list of commercially available bismaleimide products from various manufacturers is given in [6]. In this study we will concentrate on Matrimid® 5292, by Vantico (spin off of CIBA-GEIGY). Future work will include more products and comparisons between these products.

A. Matrimid® 5292

Matrimid® 5292 is a two-component bismaleimide system which when combined and used in controlled environment is suitable for vacuum impregnation. Table I gives the properties as provided by the manufacturer. For comparison the epoxy CTD-101K, which Fermilab is currently using to impregnate the Nb₃Sn dipole models, has a viscosity of about 500 cps at 60 °C with a potlife of about 40 hrs [7].

TABLE I

EFFECT OF TEMPERATURE ON THE VISCOSITY AND POTLIFE OF MATRIMID.

THE DATA WAS PROVIDED BY THE MANUFACTURER

Temperature, °C	Viscosity, cps	Potlife, min
75	5000	> 1000
100	800	1000
125	10	100
> 200	<10	< 1

Matrimid part A and B were mixed in proper proportions (1:1.13 pbw) in a mixer equipped with heating capability and mechanical stirrer. The components were heated to 100 °C with continuous stirring until a clear solution was obtained. The temperature has to be maintained within \pm 5 °C to obtain the polyimide solution with the right viscosity and potlife as indicated in the Table I. A temperature of 100 °C was chosen because it reduces the viscosity to about 800 cps sufficient for vacuum impregnation and gives a potlife of 17 hrs sufficient to impregnate long production magnets.

B. Sample Preparation

Ten-stack samples of cables insulated with ceramic tape similar to that used in Fermilab's Nb₃Sn dipole models [5] were reacted and then vacuum impregnated with both Matrimid® 5292 and CTD-101K. The procedure followed with the Matrimid was as follows:

- The two-part solution was mixed following the procedure outlined in the previous section.
- The resultant mixture was degassed while maintaining the temperature.
- The degassed solution was then used to vacuum impregnate the ten-stack sample. Note that the entire impregnation process was carried out at 100 °C.
- The curing schedule followed was 2 hours at 200 °C followed by 6 hours at 250 °C.

Fig. 1 shows a section of ten-stack sample impregnated with Matrimid. The sample was examined under optical microscope to check if the solution has reached all the areas in the sample. Fig. 2 shows a high-magnification picture of the cross-section confirming that impregnation was indeed complete.

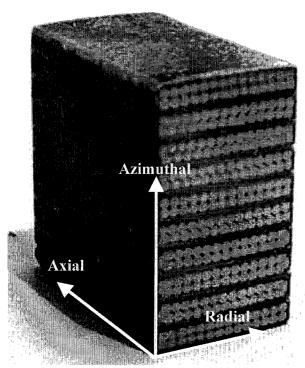


Fig. 1. Section of a ten-stack sample impregnated with Matrimid 5292.

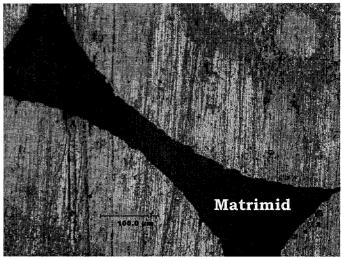


Fig. 2. Cross-section of a sample impregnated with Matrimid 5292.

The procedure followed with CTD-101K was similar to that of Matrimid except that the mixing and impregnation temperature was 60 °C and the curing schedule was 20 hrs at 125 °C. Note that the requirements for the mixing temperature are very loose, as the viscosity of the epoxy does not vary drastically at the temperature around 60 °C. The detailed procedure for epoxy impregnation can be found in [7].

C. Testing Procedure

Once the samples were impregnated, they were sectioned, polished and instrumented. Mechanical, thermal and electrical properties were obtained for samples impregnated with both matrimid and epoxy. The direction convention followed in the paper is as follows: based on Fig. 1, the vertical direction corresponds to azimuthal, direction into the paper corresponds to axial and the remaining direction corresponds to radial [8]. The following sections discuss the test results in detail.

III. MECHANICAL BEHAVIOR

The mechanical behavior of the impregnated ten-stack samples was studied both at room temperature and at 4.2 K. Tests were conducted along azimuthal and axial direction and the results obtained from samples impregnated with matrimid are compared with that of with epoxy. Strain gauges were mounted on the samples to measure the strains and a calibrated load cell was used to record the force applied on the sample. The testing fixture has been validated in the earlier tests [8].

A. Azimuthal Direction

The behavior of the overall composite in the azimuthal direction depends on all the individual constituents (conductor, insulation material and epoxy or matrimid), unlike in the axial and radial direction where the Nb₃Sn conductor mainly dominates the behavior. Figs. 3 and 4 show the test results at room temperature and at 4.2 K respectively for the composite impregnated with matrimid and epoxy. Note that the behavior of the composite is similar with both materials.

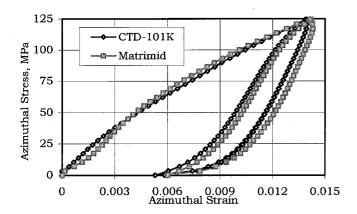


Fig. 3. Mechanical behavior of the composite in azimuthal direction at room temperature.

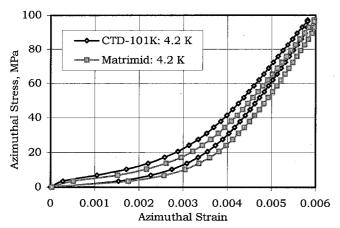


Fig. 4. Mechanical behavior of the composite in azimuthal direction at 4.2 K.

B. Axial Direction

The mechanical response of the composite in the axial direction at room temperature is shown in Fig. 5. The composite behavior is quite linear unlike in the azimuthal direction. Furthermore, the axial behavior of the composite impregnated with epoxy and matrimid is similar. This is to be expected as the behavior of the conductor dominates the composite response in axial direction.

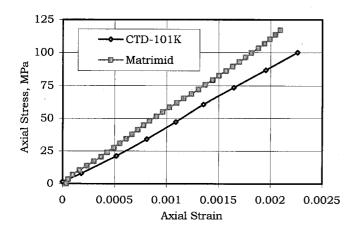


Fig. 5. Mechanical behavior of the composite in the axial direction at room temperature.

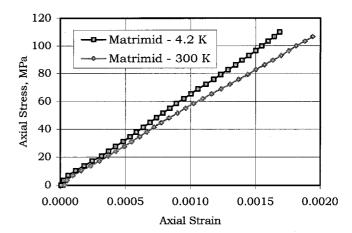


Fig. 6. Effect of temperature on the mechanical behavior of the composite impregnated with matrimid in the axial direction.

Fig. 6 shows the effect of the temperature on the mechanical response of the composite impregnated with matrimid. The stiffness of the composite increased with the decrease in temperature. Similar behavior was observed with samples impregnated with epoxy [8].

These tests reveal that the stiffness of the composite was not affected by changing the medium of impregnation from epoxy to Matrimid. This behavior is consistent with the fact that the modulus of pure CTD-101K and Matrimid is similar (5 GPa).

IV. THERMAL BEHAVIOR

The integrated thermal contraction coefficient and the thermal conductivity of the composites were measured.

A. Thermal Contraction Coefficient

The integrated thermal contraction coefficient of the composite was measured using a simple strain gage technique [9]. The temperature induced apparent strain in a sample, ε_s due to change in temperature ΔT is given by,

$$\varepsilon_{s} = \frac{\Delta R_{s}}{R F_{G}} = \left[\frac{\beta_{G}}{F_{G}} + (\alpha_{s} - \alpha_{G}) \right] \Delta T, \quad (1)$$

where $\Delta R/R$ is the unit resistance change, β_G is the thermal coefficient of resistance of grid material, $(\alpha_s - \alpha_G)$ is the difference in thermal coefficients between the sample and the grid respectively and F_G is the gage factor. If the same type of gage is installed on a standard reference material with a known thermal coefficient α_R , then

$$\varepsilon_{\rm R} = \frac{\Delta R_{\rm R}}{R \, F_{\rm G}} = \left[\frac{\beta_{\rm G}}{F_{\rm G}} + (\alpha_{\rm R} - \alpha_{\rm G}) \right] \Delta T.$$
 (2)

Subtracting the above two equations and rearranging, we get

$$\left(\alpha_{s} - \alpha_{R}\right) = \frac{\left(\varepsilon_{s} - \varepsilon_{R}\right)}{\Delta T}.$$
 (3)

Knowing α_R , ϵ_s and ϵ_R for a particular change in temperature, we can compute α_s , the integrated thermal contraction coefficient of the sample. In the present study copper was used as a reference material. Table II lists the

integrated thermal contraction coefficients of the composite from room temperature to 4.2 K for samples impregnated with both epoxy and matrimid. Note that the sample impregnated with matrimid exhibits lower thermal contraction than that impregnated with CTD-101K.

TABLE II $\begin{tabular}{ll} \textbf{INTEGRATED THERMAL CONTRACTION COEFFICIENT OF THE COMPOSITE FROM } 300\ K\ to 4.2\ K \end{tabular}$

Composite	Impregnation medium	Integrated Thermal Contraction Coefficient, µm/mm
Azimuthal Direction	Matrimid	3.1
Radial Direction	Matrimid	2.5
Azimuthal Direction	Epoxy	3.5 [8]
Radial Direction	Epoxy	2.6 [8]

B. Thermal Conductivity

The thermal conductivity measurements at 4.2 K were performed on ten-stack samples along the azimuthal direction. The cable was wrapped with a 50% overlap ceramic tape, which gives an insulation thickness of 250 μm per cable. The tests were conducted using hot—cold plate technique in collaboration with a consulting firm. The average thermal conductivity in the azimuthal direction was measured to be 0.68 W/mK for the sample impregnated with Matrimid and 0.26 W/mK for the sample impregnated with CTD 101K.

V. ELECTRICAL PROPERTIES

Ten-stack samples with alternate cables staggered (Fig. 7) were reacted and then impregnated for the purpose of high voltage tests. Fig. 7 shows a sample impregnated with epoxy. The insulation thickness between different sets of cables was maintained with reasonable accuracy as this distance affects the dielectric strength. Note that the cables have been wrapped with 50% overlap ceramic insulation tape, which amounts to an insulation thickness of 0.25 mm per cable.

The voltage was increased very slowly between two adjacent cables until the breakdown. The procedure was repeated with a different set of two adjacent cables for every pressure. Fig. 8 shows the effect of pressure on the dielectric strength of the insulation. Note that the values on the y-axis have been normalized by the initial thickness of insulation between bare cables, i.e., 0.5 mm. The data shows that the matrimid impregnated ceramic insulation has higher dielectric strength than epoxy impregnated ceramic insulation.

VI. CONCLUSIONS

The study shows that bismaleimide solutions such as matrimid can replace epoxy during vacuum impregnation of magnets. This will improve the overall lifetime of the magnet without loosing any structural or electrical integrity of the system. More tests need to be performed to perfect the impregnation procedure with Matrimid, as it is very sensitive to the temperature variation during mixing and impregnation.

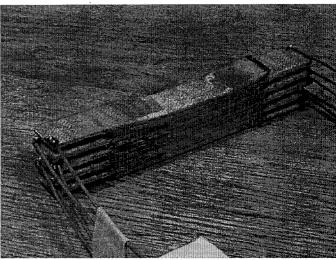


Fig. 7. Geometry of the sample used for measuring the dielectric strength.

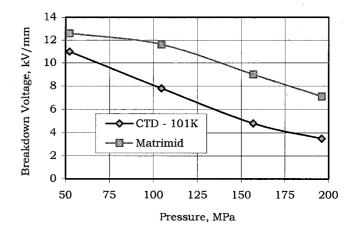


Fig. 8. Dielectric strength of the insulation as a function of pressure.

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